

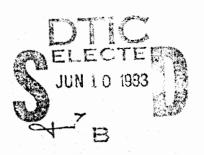
The Dust Attrition of Granular Carbon Adsorbents: Test Procedure and Typical Test Results

V. R. DEITZ, J. L. LAKSHMANAN, S. SELWYN AND J. RESING

Surface Chemistry Branch Chemistry Division

June 21, 1983





NAVAL RESEARCH LABORATORY Washington, D.C.

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A new test procedure, using the vibrate Memorandum Report 4179 has been deverabon adsorbents. The results are report the units of mgs/minute at a given vibrate the initial dust content, mg/gram of same	ting equipment preloped and applicated as a dust attitional acceleration	ied to a variety of granular rition coefficient D.A., having n. It is possible to estimate

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20. ABSTRACT (Continued)

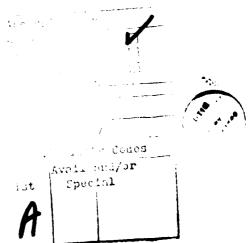
closely-sieved fractions of a carbon sample the larger particles are subjected to greater attrition than smaller particles during the test. Passage of a granular carbon through a regenerating kiln does not influence the D.A. of the particles, but the dust fraction increases slightly. The attrition behavior of adsorbent mixtures is discussed. The test procedure has been subjected to round-robin testing by the D-28 Subcommittee of ASTM.

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Thanks are due to the manufacturers and suppliers who kindly provided the samples of commercial activated carbons. The authors wish to acknowledge Mrs. Linda Hollingsworth for her most kind cooperation in preparation of the manuscript and to Mrs. Laurie Cullen for the excellent preparation of the Figures.

THE DUST ATTRITION OF GRANULAR
CARBON ADSORBENTS: TEST PROCEDURE AND
TYPICAL TEST RESULTS

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1. Introduction

Considerable experience has accumulated using the vibrating equipment previously proposed (1) to measure the dust attrition of granular adsorbent carbons. The source of the carbons has been varied widely, the parameters of the measurements have been extended to include many practical situations, and the results can now be interpreted in terms of a rate of dust attrition and a measure of the original dust content present in the sample of charcoal. The duration of the test period has been shortened to six 10-minute periods on the vibrator, a factor of some practical value for control laboratories.

The procedure to determine the rate of dust formation while passing air flows through beds of granular carbons is based on two relevant factors observed in plant operations: (1) the characteristic structural vibrations in plant scale equipment (motors, fans, etc.) that are transmitted to the charcoal particles and cause them to move across each other, and (2) the interruption or variation in the air flow that results in air pulses which can move the charcoal particles. In the test to be described a 2-inch diameter container for charcoal is vibrated at a fixed frequency and at a constant energy input manually controlled with a vibration meter in the Manuacript approved April 18, 1983.

acceleration mode. Simultaneously, an air flow is applied which exits through a particulate-grade glass filter. The quantity of dust deposited on the filter is then determined.

The need for an evaluation of dust attrition in gas-solid and liquid-solid applications has developed because of the increased use of charcoal and other solid adsorbents as impregnation—and catalyst—supports.

Moreover, in a particle—to—particle wear process leading to dust only, it is important to determine how much impregnant or catalyst would be carried away in the dust. The formation of dust in the proposed vibration equipment avoids particle breakdown as in the ''ball and pan hardness'' test or in the ''stirring test'' (1). The gentle motion among the particles having rough surfaces generates the frictional forces between particles that leads to the dust formation. The extent of motion can be increased by increase of the applied acceleration.

2. Experimental

The vibrator, Figure 1, is made of 3 aluminum cylinders (1.5 inches high, 2.5 inches diameter with a 0.25 inch wall) vertically aligned. The top cylinder, 3, holds the sample and has a fine mesh wire cloth cemented to the bottom, 4, through which the dust may fall. The dust is collected on a weighed Type A/E glass fiber filter, 6, positioned between the middle and bottom cylinders, 5 and 7. A steady downward air flow of 7 L/Min is maintained in the bottom exhaust section, 7.

The assembly is mounted on a vibration table, 9, (40 watts at 115v, 60 hertz) and the various sections clamped together with 6 mm 0.D. tie rods.

The vibration current is held constant to produce an acceleration value of 4g. After a 10-minute vibration period, the assembly is unclamped and the

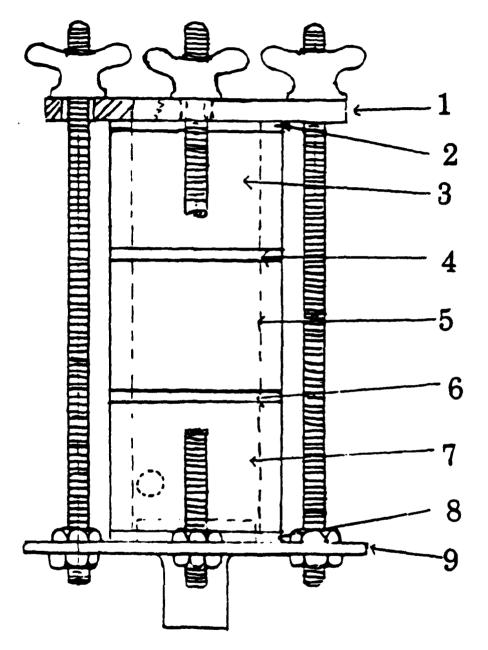


Fig. 1 - Sketch of the test unit to determine the dust attrition of granular materials.

increase weight of the glass fiber filter is determined. The procedure is then repeated.

3. Calculation Procedure for DA and DU

The quantities of dust collected at six consecutive time intervals are arranged as in Table 1A, where W_i are the weights of dust determined at the designated intervals i. Using the five intervals after the first, the slope of the regression line in a plot of integrated time, t_i , (X-coordinate) and integrated dust, W_i , (Y-coordinate) is calculated. The value of DA is defined as the slope of the regression line. The initial dust content, DU, is determined as the difference W_i - (10 x DA) mgs. in the sample.

Table 1A: Procedure to be followed in DA and DU determination.

Interval (min)	Dust (mg)	Integrated time (min)	Integrated dust (mg)
0-10	W ₁	10	V ₁
10-20	٧,	10	٧,
20-30	٧,	20	₩,÷₩,
30-40	₩,	30	₩,+₩,+₩,
40-50	٧.	f 40 f	¥,+¥,+¥,+¥,
50-60	٧,	50	W.+W.+W.+W.+W.

DA = slope of regression line of the 5 points: 2 Wi, 2 ti

 $DU = W_1 - (10 \text{ r DA}) \text{ per sample}$

Table 1B: Example of procedure for Whetlerite Lot 2-217

Weight of sample = 28.297 gram Volume of sample = 50. ml.

Interval (min)	Dust (mg)	Integrated time (min)	Integrated dust (mg)
0-10	4.70	10	4.70
10-20	3.68	10	3.68
20-30	2.40	20	6.08
30-40	3.19	30	9,27
40-50	2.82	40	12,09
50-60	2.63	50	14.72

DA = 0.281 mg/min

DU = 4.70-2.81 = 1.89 mg

DU/g = .0668 mg/gram charcoal

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4. Variation with Opening of Retaining Screen

The opening of the retaining screen furnishes the upper limit in the definition of ''dust''. The lower limit on particle size is fixed by the Type A/E glass fiber filter rated as 99.9% efficiency (0.3 μ m) by the dioctyl phthalate penetration test (DOP). The two examples (Table 2) indicate little difference in using the opening of a No. 60 or No. 100 U.S. standard sieve. The dust particles are, therefore, bracketed between <0.149 mm and >0.03 μ m.

Table 2: Variation in opening of retaining screen (acceleration 4g)

	\	coefficient 48)		•
Screen	opening (mm)	Sample weight (g)	DA (mg)	DU mg/g. charcoal
	Round	Robin Sample 3 ~	Subsample 12	
No. 60	0.250	32.68	1.43	2.0
100	0.149	33.02	1.39	2.4
60	0.250	31.30	1.31	2.1
48	0.295	32,33	1.57	0.72
		mean	1.42	1.80
		std. dev.	0.11	0.74
	1 ,	DARCO Granular C	arbon	
60	0.250	22.19	0.132	.164
60	0.250	21.56	0.126	.238
100	0.149	22.42	.145	.154
100	0.149	22.02	.139	.139
		mean	.136	.174
		std. dev.	.008	.044

The particle size distribution of the dust that is formed appears to be in a size range independent of the opening of the retaining sieve on which the carbon sample is laced.

5. Dependence on Magnitude of Acceleration

The variation of attrition with different accelerations has been observed (2) for values between 2g and 7g. The DA was always found to increase directly with the magnitude of the acceleration even for extended times on the vibrator. The choice of 4g was used as a compromise for charcoals that have small or large attrition. Typical behavior is given in Table 3 and Figure 2.

Table 3: Variation with Acceleration Applied to Vibrator

Acceleration	DA	DU/gran
ı	Coal Base Charcoal-1	•
2 g	.262	(8.18)
3g	.296	0.804
4 g	.487	0.170
	Coal Base Charcoal-2	
2 g	Coal Base Charcoal-2 0.112	0.285
2 g 3 g		0.285

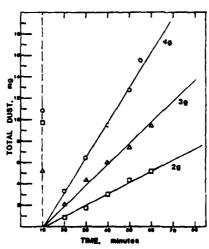


Fig. 2. - Sum of dust formed over the time interval 20 minutes to 60 minutes versus time. Each sample run at different accelerations.

6. Rate of Attrition and Extended Times on the Vibrator

The dust collected originates in the motion between individual particles subjected to frictional forces. The particle boundaries are very rough and the particles wear in a brittle manner. In this respect the behavior is similar to many ceramics. Experimentally, the strength of porous ceramics decreases nearly exponentially with increase in the volume fraction of pores (3). Hence, highly porous charcoal particles can be expected to show a similar correlation and the factors that increase the friction between the particles should likewise increase the attrition, i.e. the dust formed.

The behavior of two coal base charcoals is shown in Figure 3 where the decrease in the rate of attrition is plotted logarithmically. The behavior extends over the period between 10 to 60 minutes and furnishes the relationship used in the present attrition test.

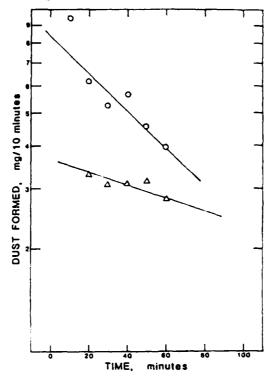


Fig. 3 - The decrease in the rate of attrition after the first 10 minutes.

For extended times on the vibrator beyond 60 minutes, the attrition process to form dust continues but at a decreased rate. The particular charcoal (Figure 4) was one that was included in the ASTM, D-28, Round Robin testing as sample No. 3, subsample 12. It is obvious that a granular carbon cannot be ''de-dusted'' since any removal operation itself generates dust continuously. The magnitudes of DA and the total dust formed are summarized in Table 4 for periods up to 420 minutes.

Table 4: Behavior of DA with Extended Times on the Vibrator (Round Robin No. 3, subsample 12)

Period	W	∑ W	DA mg/min	
0-10	12.9	12.9	1.29	
10-60	30.1	43.0	0.57	
70-120	32.6	75.6	.51	
130-180	31.6	107.2	.53	
190-240	24.0	131.2	.38	
250-300	19.4	150.6	.25	
310-360	16.7	167.3	.28	
370-420	14.9	182.2	.23	

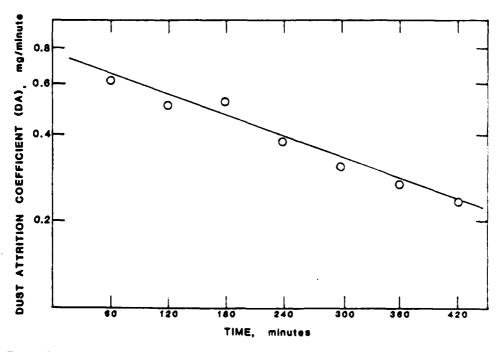


Fig. 4 - Log decrease in DA with extended periods on the vibrator.

7. Variation with Sources of Charcoals

The main objective of the dust attrition test was to differentiate among charcoals obtained from various sources. Although the described procedure does need a correlation with large scale plant experience, the test results to date align different charcoals in a plausible sequence of dust formation covering over a 80-fold range, see Table 5. The DA values are reproducible, but the estimated initial dust content, DU/gram, depends on several uncontrollable factors. There are many nooks and crannies in the boundaries of charcoal granules that can hold small dust particles. It is not known to what degree these may be released when the sample is vibrated and the particles carried away by the air flow. As an example 59.2 mg of dust (through a No. 100 sieve) was added to a coal base charcoal (12 x 30). The results (Table 6) show a large deficiency in the dust content determined. Apparently, the added dust was retained within the crevices of the charcoal and is slowly shaken free. It required about 20 minutes on the vibrator to remove 90% of the dust added. It is quite plausible that the added dust may be held by electrostatic forces where a low humidity, for example, is present.

Table 5: Variation with Charcoals from Different Sources

	DA mg/min	DU mg/gram	
Whetlerite 2-217	0.271	.0048	
	0.281	.0668	
	0.286	.0706	
Aged ASC Charcoal	0.195	.0230	
	0.227	.0531	
TEDA ASC Charcoal	0.323	.0848	
	0.343	.0547	
CAL Carbon (Service)	1.156	.489	
	1.133	.692	
Bone Char (New)	0.63	.308	
Bone Char (Service)	0.0168	.0250	
]	.0264	.0235	

Table 5: Variation with Charcoals from Different Sources (Cont'd)

	DA (mg)	DU mg/gram
Coconut shell, A	.018	.0046
Coconut shell, B	.059	.037
Coconut shell, C	.088	.060
Coconut shell, D	.017	.143
1/16th Extrudate, new	.223	.0540
	.251	.0525
1/8th Extrudate, new	.136	.042
	.119	.051
S and S, coal base	.89	.150
S and S, coal base	1.64	.192

Table 6: Attrition Test with Added Dust (i.e. (No.100 Sieve) to a Coal Base Charcoal

Time (min)	₩ (mġ)	10 (mg)	\[\sum_{\text{20}}^{60} \]
10	47.6	_	55.9
20	8.24	8.24	33.9
30	5.32	13.5	5.32
40	4.84	18.4	10.2
50	4.67	23.1	14.8
60	3,99	27.1	18.8
DA	0.53	0.47	0.45
DU	(59.2 added)	42.9	51.3

8. Variation with Particle Size

The dust attrition test has been studied with respect to particle size, the latter determined from the average openings of the testing sieves used to isolate the different sizes. The separation operation into different particle sizes produces a bias in the sample, since the operation subjects the particles to a redistribution of particles and dust. However, the results for three different charcoals demonstrate that the larger particles in a charcoal suffer more attrition than do smaller particles. It may seem, Figure 5, that the original 12 x 30 coal base charcoal had a larger DA than

any of the sieve fractions. Also, the estimated initial dust contents (Table 7) of the sieve fractions varied in the same sequence. It is of interest that the large particles indicated a greater initial dust content than did the smaller particles. However, the opposite behavior was found for a PAC coal-base charcoal where the observed dust content in the smaller particle was greater than that in the larger particles.

Table 7: Dust Attrition of Sieve Fractions Removed by Sieving from a Coal Base Charcoal (determined at 4g with 50 ml samples)

Sieve Fraction (U.S. Standard)	Average Opening mm	DA mg/min	DU/g mg/g sample
through 12-on 14	1.55	2.56	.080
through 14-on 16	1.30	1.42	.047
through 16-on 18	1.10	0.58	.019
through 18-on 20	0.92	0.71	.024
through 20-on 25	0.77	0.54	.018
through 25-on 30	0.65	1.10	.038

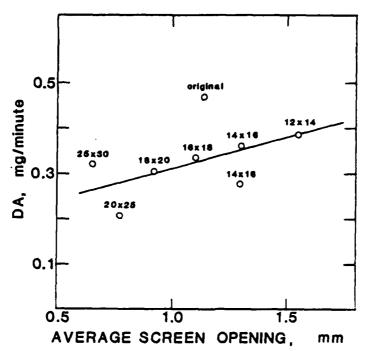


Fig. 5 - Dependence of DA on the fractions removed by sieving from a coal-base charcoal.

The behavior of sieve fractions from a coconut shell charcoal (G 210), see Table 8, may be compared to the four samples (8x16) given in Table 5. The range in DA among the sieve fractions was .011 to 0.33; the range among the four samples was .017 to .088. This behavior establishes that the attrition for coconut charcoals are minimum relative to all other gas adsorbent charcoals that have been so far examined.

Table 8: Dust Attrition of Sieve Fractions from a Coconut Shell Charcoal (NACAR G 210)

Sieve Fraction (U.S. Standard)	Average Opening mm	DA mg/min	DU mg/g
through 8-on 10	2.19	.035 .031 mean .033	.0180 .0096 .014
through 10-on 12	1.84	.039 .020 .022 .024 mean .026	.0050 .0118 .0156 .0071 .0099
through 12-on 14	1.55	.026 .029 mean .027	.026 .162 .094
through 12-on 16	1.44	.010 .012 mean .011	.103 .014 .059

The dust attrition data of five sieve fractions removed from a PAC coal base charcoal are given in Table 9. Again, the sieve fractions have DA values less than the original charcoal.

Table 9: Dust Attrition of Sieve Fractions from PAC Charcoal.

Sieve Fractions	Average Opening	DA mg/min	DU mg/g sample
through 12-on 16	1.44	0.27 0.26	.016 .036
through 16-on 20	1.02	.51 .45	.324 .333
through 20-on 30	0.715	.36 .38	.28
through 30-on 40	0.505	.28	.48 .50
through 40-on 50	0.358	.27 .25	1.23 0.57
Original		. 56	0.131

9. Behavior in a Regeneration Kiln

The mechanical integrity of granular carbons during kilning operations can be discussed from various points of view. One of the critical factors, of course, in any evaluation is the collection of a representative sample. Good samples were kindly supplied by a refinery using granular CAL carbon.

The results for evaluating dust attrition are summarized in Table 10. Within the experimental uncertainty no change in DA was observed. Good reproducibility may be noted for the two samples after kilning. The increase in dust content (per gram of sample) was definite: - 0.389 before the kiln to 0.516 mg/g. sample after the kiln. This is a good indication

that the kiln in this example (Herreshof char kiln) contributed to the dust formation.

Table 10: Dust Attrition of a Service CAL Carbon.

	Before Kiln	After	Kiln
DA, mg/min	1.02	1.05	1.00
Coeff. of variation		.996	.992
DU/gram	0.389	0.505	0.528
W ₁	21.31	24.99	25.15

10. Binary Mixtures of a Bone Char and a CAL Carbon

There is current interest in the use of mixtures of bone char and CAL carbon in the decolorization of refinery liquors (4,5). The above two adsorbents do have widely different attrition behaviors and might possibly interact during the test procedure to change the DA values for mixtures. Several binary mixtures have been examined and, as indicated in Figure 6, there are deviations from the arithmetic averages calculated for each mixture and given by the dotted line.

Good reproducibility in duplicate determinations may be encouraged by taking twice the amount of sample and making a subdivision by careful coning and quartering. The opposite quadrants in quartering were then combined. The results obtained for the binary mixtures (Table 11) were obtained in this manner.

Table 11: Attrition Behavior of Binary Mixtures of a Service Bone Char and a Service CAL Carbon.

Mixtures			
Ca1	Bone char Vol. %	DA mg/min	DU mg/g_sample
100		1.16 1.13	0.49
75	25	1.02 1.03 0.94 0.96	0.39 0.27 0.35 0.39
50	50	0.78 0.71	0.38
25	75	0.48 0.48	0.18 0.19
0	100	0.17 0.26	.025 .024

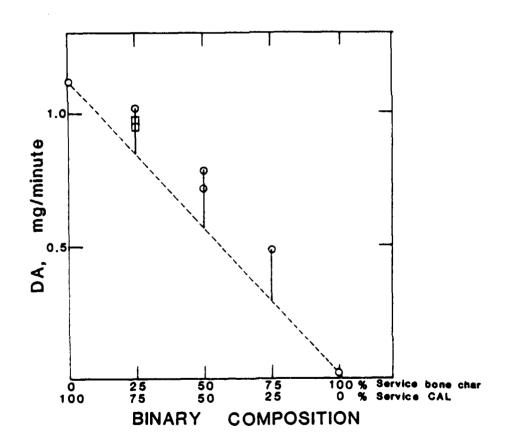


Fig. 6 - Dust Attrition, DA, of Binary Mixtures of a Service Bone Char and a Service CAL Carbon

11. Reduction in the Attrition of Granular Carbons

The dust attrition of a given material can be considerably reduced by a pre-attrition process. A vigorous fluid-flow treatment in a closed loop has been used and an example is shown in Figure 7. The DA of the initial material, a coconut shell charcoal, was determined to be 0.28 mg/min. In the pre-attrition process the corners and edges were converted to visually curved surfaces and the DA value was reduced to .009 mg/min. When the time on the vibrator of the original material was extended from 60 to 360 minutes, there was a measurable decrease in the slopes (Figure 7).

An interesting behavior was observed when a charcoal sample was tightly packed in a small cloth bag and placed in the sample space of the vibrator.

The openings of the cloth were 0.1 to 0.23 mm; for comparison, the openings of a No. 100 U.S. Sieve are normally 0.149 mm. It was found that the dust

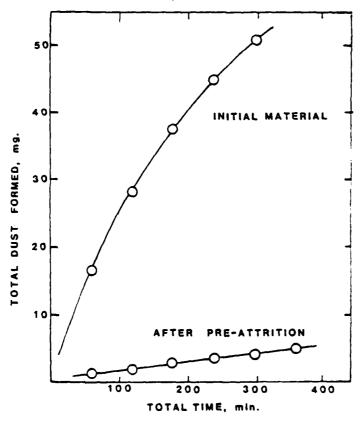


Fig. 7 - Reduction of Dust Attrition in a Special Charcoal.

attrition was markedly reduced. When the closure of the bag was relaxed and the particles assumed a loose packing, the DA increased to its previous determined value. The above observation identifies the source of the dust as due to a particle-to-particle wear process.

12. Concluding Remarks

The proposed test procedure for dust attrition has been successful in evaluating samples of granular activated carbons with good reproducibility.

The requirements of a test procedure for technical control purposes have been realized, namely:

- 1. The experimental operation should be relevant to the adsorbent application.
- 2. The results must be adequately precise.
- The test should be conducted with minimum technician time and maximum simplicity.
- 4. The maintenance of the equipment should be practical in time and capital investment.

The proposed ASTM, Standard Method of Test for Dusting Attrition of Granular Activated Carbons, has progressed to Draft No. 3 (22 February 1983) and will be submitted for a vote by the D-28 committee members shortly. A copy is given in Appendix 1. In addition, the test results are capable of aligning different charcoals in a sequence that agrees at least qualitatively with practical experience.

When using impregnated—and catalyst—carbons, the composition of the dust collected during an attrition test can be a measure of the loss of the impregnant and or catalyst. Also, with systematic applied pre-attrition, the measurement can be used to determine the depth of an impregnation into carbon particles.

13. References

- Deitz, Victor R. ''The Dusting Attrition of Granular Carbon Adsorbents' Proc. Sugar Industry Technologiest, Inc. 37, 473-488 (1978) Paper 425.
- Deitz, Victor R. and Pongpat, Poonsuk 'Dust Formation by Attrition of Granular Activated Carbons' NRL Memorandum Report 4179, 37 pp, 27 February 1980.
- 3. Ryshkewitch, E. J. Am. Ceram. Soc. <u>36</u>, 65-68 (1953).
- 4. Barton, W.V. and Knebel, W.J., ''The First Process Implementation of the Canesorb Carbon/Bone Char System: Atlantic Sugar'', S.I.T. Paper 480 Sugar Industry Technologists 41, 189-233 (1982).
- 5. Sulick, D.J., ''Regeneration of an Admixture of Bone Char/Granular Carbon in Vertical Retort Kilns'', S.I.T. Paper 481, Sugar Industry Technologists 41, 234-242 (1982).

14. Appendix: Proposed ASTM Standard Method of Test for Dusting Attrition of Granular Activated Carbon (Draft No. 3 of 22 February 1983)

1. Scope

1.1 This method covers the determination of the dusting attrition of granular activated carbons. For the purposes of the test the dust attrition coefficient, DA, is defined as the weight (or calculated volume) of dust per unit time collected on a preweighed glass fiber filter in a given vibrating device during a designated time per unit weight of carbon. The initial dust content of the sample may also be determined. Granular activated carbon is defined as a minimum of 90 percent being larger than 80 mesh (0.18 mm), see ASTM Method D2862.

2. Applicable Documents

- D2652. Standard Definitions of Terms Relating to
 Activated Carbon
- D2854. Standard Method of Test for the Apparent

 Density of Activated Carbon
- D2867. Standard Method of Test for Moisture in
 Activated Carbon
- D3195. Recommended Practice for Rotometer Calibration
- E 300. Recommended Practice for Sampling Industrial
 Chemicals

3. Summary of Method

3.1 A specified volume of known weight of the granular activated carbon is placed in the prescribed sample holder and vibrated at constant acceleration, "g", for a specified time. The dust is carried by an air stream passing through the vibrating sample and is then collected on a

preweighed glass fiber filter. The quantities of dust collected in six 10minute intervals are determined by weighings on an analytical balance.

4. Significance and Uses

4.1 Three forces can mechanically degrade a granular activated carbon: impact, crushing, and attrition. Of these three, attrition, or abrasion, is the most common cause of dust formation in actual service. Published test procedures to determine the 'hardness' of activated carbons produce results which in general can not be correlated with field experience. For example, the ball-pan hardness test applies all three forces to the sample in a variable manner determined by the size, shape and density of the particles. The 'stirring bar' abrasion test measures attrition so long as the particle size is smaller than 12 mesh. There is some evidence, however, that the results of this method are influenced by particle geometry. The method described herein measures the effect of friction forces between vibrating or moving particles and may be only slightly dependent on particle size, shape and density effects.

5. Apparatus

- 5.1 Vibrating table capable of providing an acceleration of 4
 ''g'' at 60 Hz. A Buffalo Dental Manufacturing Company Vibrator, Model No.

 1, rated 40 watts at 115 volts, 60 Hz has been found satisfactory.
 - 5.2 Test Cell, as shown in Figure 1.
- 5.3 Piezoelectric accelerometer capable of measuring accelerations of 4 g at 60 Hz with an output signal between 5 and 20 mV/g. An Endevco Model 2251 Accelerometer has been found to be satisfactory.
- 5.4 Signal Conditioner, to interface the accelerometer with a d.c. millivolt meter, capable of producing a linear output voltage from 0 to

100 mv proportional to the acceleration. An Endevco Model 4416 Signal Conditioner, battery operated, has been found to be satisfactory.

- 5.5 Voltimeter, 0-100 mV dc, Keithley 179 digital multimeter has been found satisfactory.
 - 5.6 Ammeter, a.c., 0 to 1000 ma accurate to 1 ma.
 - 5.7 Variable transformer, 0 to 120 volts.
 - 5.8 Timer Control, 0 to 120 minutes.
- 5.9 Isolation pad, cross-ribbed, 9.5mm (3/8'') thick x 100mm (3.94'') square constructed from 45 durometer neoprene rubber.
- 5.10 Flowmeter with flow control valve having a capacity from 1 to 12 liters per minute.
- 5.10 Glass fiber filters, 63.5mm diameter having an efficiency of 99.9% for 0.3µm particles as measured by Dioctyl Phthalate Penetration tests. Several commercial glass fiber filters have been found satisfactory.
 - 5.12 Graduated cylinder, 50ml capacity.
 - 5.13 Analytical balance, capable of weighing to 0.1mg.

6. Sampling

6.1 Guidance in sampling granular activated carbon is given in Recommended Practice E 300.

7. Apparatus Assembly

- 7.1 The test cell is assembled as shown in Figure 1. Components of the test cell which must be fabricated are shown in Figure 3.
- 7.2 The wire cloth for the bottom of the top section is glued between two rubber gaskets and this assembly is then glued to the bottom of the section to form the sample holder.

- 7.3 Mount the accelerometer at the center of the cover plate, either by cement or by using the cap screw (a hole threaded for #52 is specified).
- 7.4 Place the vibrator test assembly on the vibration isolation pad so that the cover plate of the test cell is level.
- 7.5 Connect the accelerometer lead to the signal conditioner, then connect the output of the signal conditioner to the microvolt meter (Fig. 2).
- 7.6 Connect the vibrator to the variable transformer and connect the transformer to a timer control with the milliammeter in series (Fig. 2).
- 7.7 Connect the air outlet of the attrition test cell to the flowmeter, and connect the flowmeter to a vacuum source (Fig 2).

8. Test Procedure

- 8.1 Measure out 50ml of the sample into a tared, graduated cylinder using the method and apparatus described in ASTM D2854 \$23d weight to the nearest 0.1g. Reproducibility in duplicate determinations may be improved by taking twice the volume of sample (100 ml) and making a subdivision by careful coming and quartering. The opposite quadrants after quartering are then combined for the test sample.
- 8.2 Calculate the apparent density of the sample using the method of ASTM D2854.
- 8.3 Quantitatively transfer the sample into the sample holder section of the test cell.
- 8.4 Weigh the glass filter element and the dust to 0.1 mg or better.

- 8.5 Insert the weighed glass filter between the two flat teflon gaskets and place in position on top of the exhaust section of the test cell. Handle the glass filter with care to avoid tearing.
 - 8.6 Place the spacer section of the test cell in position.
 - 8.7 Place the sample holder of the test cell in position.
- 8.8 Place a rubber gasket on top of the sample holder, then position the cover plate through the four threaded rods.
- 8.9 Place O-rings or flat washers on the threaded rods and secure the test cell assembly to the vibrator table by threading the wing nuts to finger tightness.
- 8.10 With the vibrator control always set at the HIGH range position, set the timer control to 10 minutes.
- 8.11 Start the air flowing, then adjust to draw 7 liters per minute of air through the sample.
- 8.12 Increase the voltage to the vibrator from zero to produce an acceleration of 4.0 "g" as measured by the accelerometer and signal conditioner. Alternatively, the vibrator current may be adjusted to the value furnished by a calibration using the accelerometer.
 - 8.13 Vibrate the sample for 10 minutes.
- 8.14 Remove the wing nuts, cover plate, sample holder and spacer section of the test cell to expose the filter element.
 - 8.15 Carefully remove and weigh the glass filter to 0.1 mg.
 - 8.16 Insert a second weighed glass filter.
- 8.17 Repeat steps 8.4 to 8.16 for a total of six 10-minute vibration intervals.

9. Calculations

9.1 Calculate the total dust collected during the following designated time intervals:

Interval (min)	Weight (mg)	Integrated time (min)	Integrated Weight (mg)
0-10 10-20 20-30 30-40 40-50 50-60	W1 W2 W3 W4 W4	10 20 30 40 50	W ₁ W ₂ W ₃ + W ₅ W ₂ + W ₃ + W ₄ W ₂ + W ₃ + W ₄ + W ₅ W ₂ + W ₃ + W ₄ + W ₅

- 9.2 Using the integrated time intervals as X-coordinate and the corresponding total dust as Y-coordinate, and excluding the first 10-minute interval, calculate a least squares linear regression on the five pairs of X_i , Y_i where i = 2, 3, 4, 5, 6.
- 9.3 The slope is the dust attrition, <u>DA</u>, in mg/minute; calculate the correlation coefficient.
 - 9.4 Calculate the initial dust content, $w_1 10$ DA.
- 9.5 Calculate the dust attrition coefficient by volume, DA(V) from the equation

 $DA(V) = DA(W)/apparent \ density, \ \mu \ell$ where apparent density is expressed as mg per $\mu \ell.$

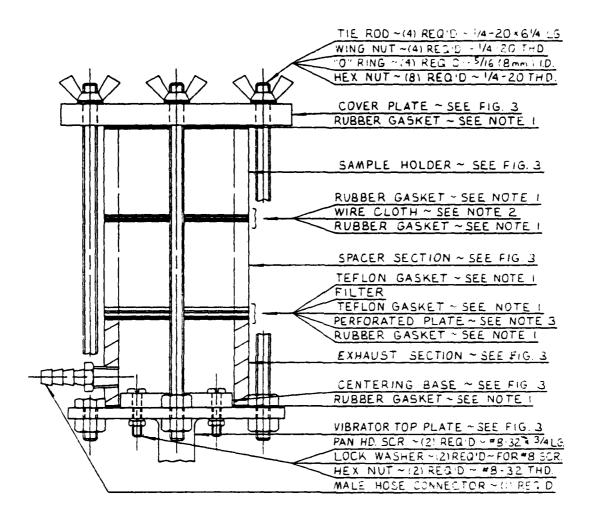
10. Report

Report the name of the carbon supplier, the grade or type designation of the sample, the lot and batch number, the dust attrition coefficients, the initial dust, and the name of the agency and technician making the test.

11. Precision and Accuracy

To be determined by round robin testing.

- NOTE 1. The apparatus will resonate at frequencies near 55 cps. If the frequency of the power source varies appreciably, it may be necessary to provide frequency control at 60 cps.
- NOTE 2. If the voltage of the power source varies by more than 2 percent, it is advisable to employ a voltage stabilizer or regulator.
- NOTE 3. It has been found that during continuous use the vibrator becomes warm enough to influence the g-value. This may be prevented by placing the isolation pad and vibrator within a shallow tray (for example, a photographic development tray) and adding a few pieces of dry ice around the inside wall to cool the air in contact with the vibrator.



NOTES .

- 1. 2" (50.9 mm.) 1.D. × 21/2"(63.5 mm.) O.D. × 1/16 (1.6 mm.) THICK.
- 2. ASTM E II WIRE CLOTH, 149 µm, STAINLESS WIRE, 2 12"(63.5 mm) 0:4.
- 3. 37% OPEN AREA, FABRICATED FROM 24 GAUGE STAINLESS STEEL WITH 0.045" DIA. HOLES ON 0.066" CENTERS, SQUARE GRID. 21/2"(63.5 mm.) DIA

Figure 1: Test cell for dusting attrition of granular adsorbents.

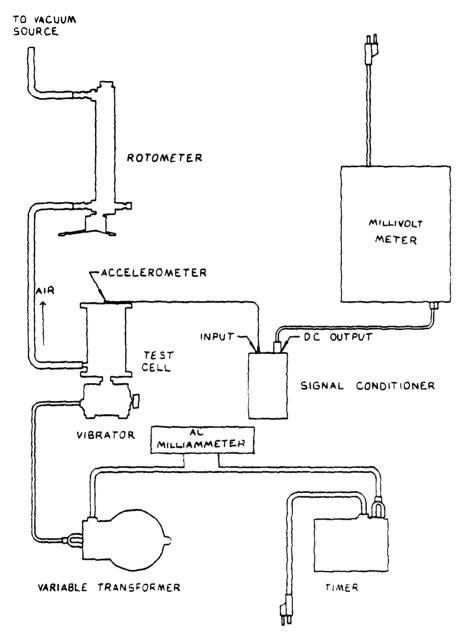


Figure 2: Assembly indicating location of vibrator and accessories.

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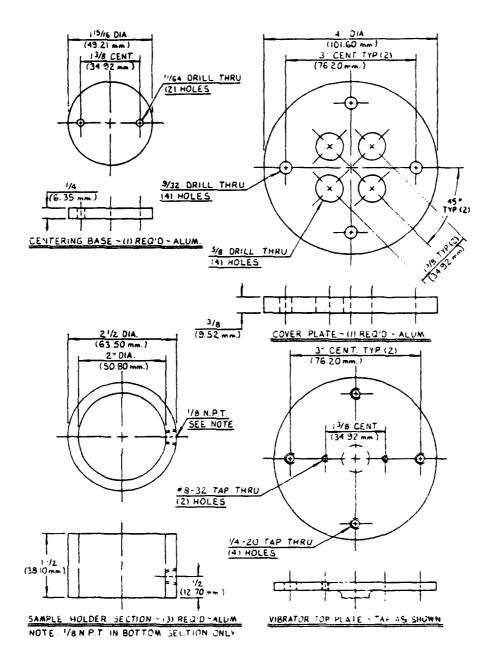


Figure 3: Construction details of vibrator parts.